Synthesis and Characterization of Various Morphology Based Heterojunction Nanocomposite for Supercapacitor Application

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By

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Final synopsis

1. Introduction

With the rapid development of sustainable and renewable energy storage super capacitor (SC) device with excellent specific capacitance and high energy density are rise various challenging issues need to be address for energy consumption in daily needs [1-2]. Supercapacitors are one of the prominent energy storage devices among different energy storage devices due to their excellent energy density, long cycle life, fast charging-discharging capacity, and low maintenance cost with many advantages over batteries and also occupies vast scope of usage in various purposes [3]. The major demerit of the supercapacitor is that they have low energy density. Therefore, there is a need to enhance the energy density and voltage window without losing the power density and cyclic stability. From the energy density relation $E=1/2CV^2$, by increasing the voltage window (V) and specific capacitance (Csp) may enhance the energy density (E) of the supercapacitor.

An alternative approach to improve energy density is to develop hybrid electrochemical capacitors (so called asymmetric supercapacitors, or ASCs), which can also provide a wider operating potential window compared to symmetric supercapacitors. The use of redox active materials enhance substantially the energy density of supercapacitors due to their much higher specific capacitance 10–100 times more than carbonaceous materials. In case of aqueous-based supercapacitor devices, the voltage window of symmetric supercapacitor (SSC) is normally restricted up to 1.0 V but for asymmetric supercapacitor (ASC) it can be extended up to 2 V by using redox-active material cathode and activated carbon anode. The active carbon as anode and redox active material as cathode provide the high hydrogen and oxygen over potential respectively which enhances the potential window of aqueous-based ASC.

Mainly, supercapacitor can be categorized into i) electrical double layer capacitors (EDLCs) and ii) pseudocapacitors (PC) based on the various charge storage mechanisms [4-5]. In

general, energy storage process shows electrostatic behavior in EDLCs and separation of charges takes place at the interface of electrode and electrolyte. Conventional carbon-based materials like activated carbon, carbon nanotubes and graphene etc,[6-8] are commonly used in EDLCs whereas in pseudocapacitors charge storage mechanism is faradic in nature i.e., electron charge transfer takes place between electrode and electrolyte. Conducting polymers are preferred as a pseudocapacitors electrode materials because of their significant advantages in intrinsic flexibility and high electrical conductivity compared to other materials which makes them perfect candidates for high-performance flexible SCs. For this purpose, conducting polymers like polyaniline, polypyrrole, polythiophene [9-12] and metal oxides like RuO_2, MnO_2, NiO, Co_3O_4, ZnO and Al_2O_3 [13-17] are used to develop desirable and flexible storage devices.

Among various transition metal oxide pseudocapacitive materials, Zinc Oxide and Nickel Oxide (NZ) is also considered as one of the most important semiconducting and electrode materials for high performance specific capacitor as it owes high specific capacitance, excellent stability, easy preparation, and various morphologies [18-20]. Thus, ZnO is widely used to increase the electrical conductivity as well as prevent the aggregation of active material. The morphology of ZnO such as nanospheres, nanotube and nanowire plays an important role in enhancement in the electrochemical properties. Nevertheless, the pure metal oxide suffers from relatively low conductivity and weak electrochemical stability which is incapable for the requirement of high specific capacitance supercapacitors [21-22]. To overcome these drawbacks and to improve the electrochemical performance of pseudocapacitive materials, several approaches such as nanostructure based materials have been successfully developed. One of the emerging attractive concepts is to fabricate heterojunction nanostructured nanocomposites. It has been demonstrated that electrode materials with heterogeneous nanostructure could gain high specific area, short ion diffusion path and increased active sites.

And also conductivity of the sample can be increased by incorporation of conducting additive material [23-24].

One of the more intriguing developing ideas is to create unique actinomorphic flower-like SnO_2/ZnO nanostructured morphology, these morphological based electrode materials exhibited a high specific area, short ion diffusion path, and more active sites. In addition, the electrical conductivity of the material can be improved by using a conducting additional material [25]. The ZnO is also used utilised to improve electrical conductivity and avoid active material aggregation. The electrochemical characteristics of SnO_2/ZnO are enhanced due to significant change in their morphology such as nanospheres, nanotubes, and nanowires [26-27]. However, SnO_2 have a lower cycle life than ZnO doped metal oxides because of the expand and contract significantly when charging and discharging process as result drop in the specific capacitance. Considering the properties of metal oxide, SnO_2 and ZnO, it will be important to ornament the metal oxide nanoparticle on the outer surface of SZ in which metal oxide and SZ prevents nanoflowers from swelling and contrast during the charging-discharging process [28]. Three-dimensional hierarchical honeycomb-like porous carbons (3D-HHPC) stand out because of its large surface area, ordered porous structures, high conductivity, and high efficiency, low cost, and unique natural shape [29]. Because of their abundance, many sustainable organic resources were intensively studied as substrates for the simple, effective, and inexpensive synthesis of honeycomb-like porous carbons. Ganoderma Lucidem Spore (GLS) is a mushroom that is generally referred to as Lingzhi in Chinese and it has been used as a therapeutic mushroom in traditional Chinese medicine since from long ago [30]. GLS is frequently used as a medication because of the presence of different amino acids, anti-allergic, anti-inflammatory, anti-cancer, and anti-diabetic. It can also use GLS as a biomass carbon source to make supercapacitor materials is tremendously valuable and necessary. This work introduces a new environmentally favorable, low-cost, and simple-to-use carbon source, as

well as a cost-effective and technologically unique method for carbon supercapacitor applications [31]. Therefore, recently many researchers paid much attention to achieve high-performance active electrode materials for supercapacitor. This current study would be allocated for developing a novel hybrid composite material for EDLC and pseudocapacitors.

2. Aims and objective of the present study

In order to address the research problem, the following thematic research aims and objective are formulated below;

1. To synthesize and characterization of various morphology based heterojunction nanostructured nanocomposite materials for supercapacitor applications.
2. To investigate and study the n-type and p-type semiconducting heterostructured metal oxide materials such as binary metal oxide, ternary metal oxide and bio-wasted material.
3. Incorporation of metal oxide into nanostructured materials evaluate their properties in different type of methods like hydrothermal, carbonization, etc.
    i. Self-Templated Synthesis of Hierarchical Actinomorphic Flower-like SnO_2-ZnO.
    ii. Zinc oxide incorporated with Nickel oxide (NZ) hexagonal nanorods nanocomposite.
    iii. Synthesis of hierarchical ZnO/NiO nanocomposite Wurtz-like Hexagonal Nanorods.
    iv. Self-structured 3D-Hierarchical Honeycomb-like Porous Carbon derived from Ganoderma Lucidem.
3. To characterized Nanostructured nanocomposite with help of various techniques to examine Chemical, structural, and morphology.
4. To fabricate electrode with different modes and environmentally favorable as well as a cost-effective and technologically unique method for supercapacitors applications such as flexible, high energy density all-solid-state symmetric supercapacitor (SSC).

3. The brief sketch of the proposed thesis is as follows

The fundamental goal of this thesis is to synthesis, characterization, and electrochemically performed nanostructured heterojunction nanocomposites for use in all-solid-stateSSC. Thesis contains seven chapters; the First chapter includes the introduction to metal oxide and porous carbon nanocomposite, as well as their use in supercapacitors and its application. The Second chapter discusses the numerous approaches used in the synthesis, characterization, and fabrication of heterostructured material based supercapacitors. The outcomes of the experiments carried out to achieve the study objectives are discussed in the Third to Seventh chapters. The third chapter includes the "Self-Templated One-Step Hydrothermal Synthesis of Hierarchical Actinomorphic Flower-like SnO_2-ZnO Nanorods for High-Performance Supercapacitor Applications". The fourth chapter contains "Single-Step Hydrothermal Synthesis of ZnO/NiO Hexagonal Nanorods for High-performance Supercapacitor Applications". The fifth chapter encloses "Synthesis of hierarchical ZnO/NiO nanocomposite Wurtz-like Hexagonal Nanorods via hydrothermal for high-performance symmetric Supercapacitor applications". The sixth chapter emphasizes on "self-structured 3D-Hierarchical Honeycomb-like Structured Porous Carbon derived from Ganoderma Lucidem for High-performance Supercapacitor". In the seventh chapter, the summary and conclusion are discussed, followed by the sections of references and list of research papers.

Chapter I: Introduction

The first chapter gives an overview of nanostructured metal oxide and porous materials, as well as their applications in supercapacitors. A basic description of supercapacitors, including their history, charge storage mechanism, working principal, and uses, as well as a detailed literature review and motivation to determine research goals and objectives.

Chapter II: Materials, Characterization and Fabrication Methods

The fabrication of nanostructured metal oxide composites is described in this chapter, as well as the characterization methodologies utilized to investigate material characteristics such as structural, morphological, thermal, and electrochemical properties. This section also covers the fabrication of electrode materials for supercapacitor devices of various configurations and electrolytes.

Chapter III: Self-Templated Hydrothermal Synthesis of Hierarchical Actinomorphic Flower-like morphology SnO_2-ZnO Nanorods for High-Performance Supercapacitor Application

In this chapter, we used a hydrothermal approach to make Self-Templated One-Step Hydrothermal Synthesis of Hierarchical Actinomorphic Flower-like SnO_2 Nanorods doped on Zinc Acetate Dehydrate (SZ). SnO_2 serves as the primary material, while ZnO serves as the explosive material, resulting in an actinomorphic flower-like nanorod structure. Given the properties of SnO_2 and ZnO, it will be important to adorn the outside surface of actinomorphic floral nanorods, which SnO_2 and ZnO prevent against swelling and contrast during the charging-discharging process. Furthermore, SZ with an actinomorphic flower-like nanorod structure functioned as a conductive and skeleton for the nanocomposite, which is advantageous for electron collecting and rapid transport. The electrochemical analysis reveals that the SZ-10 nanostructure has a high specific capacitance of 797.23 Fg^{-1}, which is much greater than SZ-5 (602.7 Fg^{-1}) and SnO_2 (485.1 Fg^{-1}), at a current density of 1 Ag^{-1}. Furthermore, an all-solid-state symmetric supercapacitor made with the SZ-10 electrode has a maximum ED of 45.72 $Whkg^{-1}$ and a PD of 406 Wkg^{-1}. Even after 5000 cycles, the excellent capacitance retention of 88.2% and coulombic efficiency of 98.7% is maintained. This research demonstrates the high potential of SZ actinomorphic flower-like nanorods as a high-performance supercapacitor electrode material.

Chapter IV: Single-Step Hydrothermal Synthesis of ZnO/NiO Hexagonal Nanorods for High-performance Supercapacitor Applications

The current chapter provides an overview of zinc oxide hexagonal nanorods that have been combined with nickel oxide nanostructure using a hydrothermal process. In this paper, we studied the potential uses of a novel cost-effective NiO nanoparticle combined with Zinc oxide nanostructure (NZ) in which NZ hexagonal nanorods are generated for all-solid-state symmetric supercapacitor (SSC) applications. We synthesised ZnO, NZ-5 and NZ-10, nanoparticles with NZ nanostructures for comparative investigations and characterized them utilizing various methods. The NZ-10 nanocomposite has a low charge transfer resistance (0.86) and a high specific capacitance of 665.4 Fg^{-1}, which is much greater than NZ-5 (530.7 Fg^{-1}), and ZnO (411.7 Fg^{-1}), at a current density of 1 Ag^{-1}. Furthermore, an SSC constructed with NZ-10 electrode has a maximum Energy Density of 26.73 $Whkg^{-1}$ and Power Density of 641.5 Wkg^{-1}. It also has excellent cyclic stability, with capacitance retention of 73.43 % and coulombic efficiency of 96.12 % after 5000 cycles. To validate the practical usage, the constructed SSC is successfully used to light a commercial blue LED, its glow 1 to 2 min. The excellent electrical conductivity is largely responsible for NZ-10 outstanding performance. The findings show that NZ hexagonal nanorods are a suitable supercapacitor electrode material Application.

Chapter-V: Synthesis of hierarchical ZnO/NiO nanocomposite Wurtz-like Hexagonal Nanorods via hydrothermal for high-performance symmetric Supercapacitor applications

This paper describes a simple and well-controlled synthesis of transition metal oxides based on Nickel oxide-Zinc oxide (NZ) binary nanocomposite to construct free-standing and stable hybrid Wurtz Hexagonal Nanorods (HN) via hydrothermal technique. The NZ nanocomposite was used and analyzed using several methods including as XRD, FTIR, FESEM, HRTEM, XPS, and Raman Spectroscopy. The electrochemical performance of the two electrode system symmetrical separate electrode was achieved using the NZ-15 binary nanocomposite, which had a CD of 1 Ag^{-1} and a high specific capacitance (Csp) of 933.2 Fg^{-1}. Even after 5000 cycles, the capacitance retention is 79.64 % and the coulombic efficiency is 97.24 %, indicating excellent long-term cycle stability. It had a maximum energy density (ED) of 115.8 $Whkg^{-1}$ and a power density (PD) of 1042.4 Wkg^{-1}, as well. The electrodes practical use was assessed by lighting it up with a commercial blue LED linked in series, which flashes for nearly 1 to 2 min with intermittent voltage drops. The high electrochemical performance and other results could be evidenced by the development of innovative electrode materials and hybrid wurtz HNs materials are a prominent candidate for high-energy-density supercapacitor applications.

Chapter VI: Self-structured 3D-Hierarchical Honeycomb-like Porous Carbon derived From Ganoderma Lucidem for High-performance Supercapacitor Application

In this chapter, we concentrated on making three-dimensional hierarchical honeycomb-like porous carbons (3D-HHPC) stand out due to their huge surface area, ordered porous structures, high conductivity, high efficiency, low cost, and distinctive natural form. Because of their availability, numerous sustainable organic resources have been extensively researched as substrates for the easy, effective, and low-cost synthesis of honeycomb-like porous carbon material for supercapacitor applications (SC). The primary goal of this experiment is to develop a highly efficient mechanism that is inexpensive in cost, mass-produced on a huge scale, and environmentally acceptable. We constructed a flexible solid-state SCs out of Ganoderma Lucidem porous carbon material to examine the micromorphology, pore structure, and pore size distribution of 3D-HHPC systems. At a current density (CD) of 2 Ag^{-1} using 4M KOH electrolyte, the greatest specific capacitance (Csp) of the 3D-HHPC/900 electrode was determined to be 242.6 Fg^{-1}, which is much greater than HHPC/800 (203.8 Fg^{-1}) and GLS (150.3 Fg^{-1}). The energy density (ED) of the 3D-HHPC/900 electrode is 92.38 $Whkg^{-1}$ and the power density (PD) is 1662.9 Wkg^{-1}. More importantly, even after 4000 repeated cycles, the SCs device maintains remarkable performance, with an 84.4 % capacitance retention and a coulombic efficiency of 97.6 %. Furthermore, the symmetric device utilized to glow LED for more than 30 sec to 1 min. This GLS HHPC/900 also has stable electrochemical properties at different temperatures, and this study introduces a new environmentally friendly, low-cost, and easy-to-use carbon source, as well as a cost-effective and technologically unique method for carbon supercapacitors in environmental applications.

Chapter VII: Summary and Scope for Future work

Summarizes the findings of the present investigations and the scope for further studies in this field.

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List of Journal Publications:

1. A paper entitled *"Self-Templated One-Step Hydrothermal Synthesis of Hierarchical Actinomorphic Flower-like SnO_2-ZnO Nanorods for High-Performance Supercapacitor Applications"* in **Journal of Electroanalytical Chemistry,** (2021). 900, 115741.
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2. A paper entitled *"Single-step hydrothermal synthesis of Nickel Oxide-Zinc Oxide heterostructured Material for high-performance Supercapacitor Applications "in* **Material Science in Semiconducting Processing,** (2021).
   Nagaraju Y.S, Ganesh H, Veeresh S, Vijeth H, Basappa, Devendrappa H*.

3. A paper entitled *"Synthesis of hierarchical ZnO/NiO nanocomposite Wurtz-like Hexagonal Nanorods via hydrothermal for high-performance symmetric Supercapacitor Application".*
   (Review copy submitted to journal)
   Nagaraju Y.S, Ganesh H, Veeresh S, Vijeth H, Devendrappa H*.

4. A paper entitled *"Self-Structured 3D-Hierarchical Honeycomb-like Porous Carbon derived from Ganoderma Lucidem for high-performance Supercapacitor Application".*
   (The journal process is under review)
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5. A paper entitled *"Single crystalline hierarchical SnO_2 microsphere and fluoride-mediated hollow structures for photocatalytic activity".* In **Materials Today: Proceedings** (2020). *45*, 3833-3836.
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25. A paper entitled *"Low Cost Synthesis and Characterization of Donor P3HT Polymer for Fabrication of Organic Solar Cell"* in **Material Science and Engineering "**in (2022).
D S Suresh, M Vandana S Veeresh1, H Ganesh, **Nagaraju Y.S,** H Vijeth, M Basappa and H Devendrappa *

Conferences Proceedings and Virtual Workshops:

1. Participated & presented an article entitled *"Single crystalline hierarchical SnO_2 microsphere and fluoride-mediated hollow structures for photocatalytic activity"* in ***ICNAN-2019*** Organized by Centre of Nanotechnology Research, Vellore Institute of Technology. Vellore, India during 29th November -1st December 2019.
   Nagaraju Y.S, Ganesh, H., Veeresh, S., Vandana, M., Ashokkumar, S. P., Vijeth, H., & Devendrappa, H*.

2. Participated & presented an article entitled *"Hydrothermal synthesis of highly crystalline NiO doped ZnO hexagonal shape nanorods arrays as cathode material for photocatalytic activities"* in **DAE Solid State Symposium-2019** Organized by Bhabha Atomic Research Centre, Mumbai. Indian Institute of Technology Jodhpur, Rajasthan India during 18th-22th December 2019.
   Nagaraju Y.S, Ganesh, H., Veeresh, S., Vandana, M., Ashokkumar, S. P., Vijeth, H., & Devendrappa, H*.

3. Participated & presented an article entitled *"Facile hydrothermal synthesis of Zn-doped Sno_2 for crystallographic facet-oriented polyhedral structure" in* ***ICPN-2019.*** Organized by Dept. of Physics Mangalore University, Mangalore, and Karnataka India during 19th-21st September 2019.
   Nagaraju Y.S, Ganesh, H., Veeresh, S., Vandana, M., Ashokkumar, S. P., Vijeth, H., & Devendrappa, H*.

4. Participated & presented an article entitled*"To study the synthesis and characterization of ZnO hexagonal nano cubes with hydrothermal growth and formation mechanism" in* ***ICPN-2019.*** Organized by Dept. of Physics Mangalore University, Mangalore, and Karnataka India during 19-21 September 2019.
   Nagaraju Y.S, Ganesh, H., Veeresh, S., Vandana, M., Ashokkumar, S. P., Vijeth, H., & Devendrappa, H*.

5. Participated in the "*International Symposium on Advanced Research in Physics – 2020*" organized by the Department of Physics, Bangalore University.

6. Participated in the *"One Day Online National Level Workshop on "CONSTRUCTIVISM-2021"* organized by the Department of Physics, P.C. Jabin Science College, and Hubballi.

7. Participated in the *"Advanced Materials for Energy Applications-2021",* organized by the Department of Physics, Mangalore University.

8. Participated and working as a local organizing committee member in the *"Second International Conference on Physics of Materials & Nanotechnology* ***(ICPN-2021),*** organized by the Department of Physics, Mangalore University.

Foreign Visit:

- Participated & presented an article entitled *"Incorporating waste carbon fly ash to study the annealing effects of structural and optical properties of Rock-salt morphology controlled by Electrolyte for Energy Storage Application"* in the 4th International Conference on Applied Sciences, Engineering, Technology and Management-2021 **(ICASETM 2021)** held at **Dubai** during 29-30th December 2021.

  Nagaraju Y.S, Ganesh H, Veeresh S, Vijeth H, Basappa, Devendrappa H*.

Workshops Attended:

SL. No	Organization	Period		Details of training/project undertaken
		From	To	
1	KSTA Karnataka State and Technology Academy, Bangalore	November 2017	November 2017	"Basic training in science and its applications in everyday life, as well as an exhibition".
2	CENSE Indian Institute of Science, Bangalore.	October 2019	October 2019	"INUP- basic training course about nanoparticles".
3	Dept. Of Chemistry, Mangalore University, Konaje.	03rd December 2018	08th December 2018	GIAN workshop on **"**Multifunctional Advanced Nanomaterials and their Applications"

Dr. Devendrappa H
(Research Guide)

Nagaraju YS
(Research student)